"ELUCIDATING THE SYNTHESIS, CHARACTERIZATION, AND VERSATILE APPLICATIONS OF NANOSTRUCTURES COMPRISING NICKEL OXIDE AND COPPER OXIDE"

SYNOPSIS

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Introduction

A sustainable environment in human existence refers to the creation and maintenance of a balance between human activities and the natural world, assuring the long-term well-being of both. It entails preserving resources, reducing waste and pollution, and encouraging ecological harmony. Sustainable practices seek to fulfil the demands of the current generation without harming the capacity of future generations to meet their own needs (Hazarika et al., 2022). Nanoparticles, on the other hand are incredibly tiny particles with dimensions on the nanometre scale. To put things into context, one nanometre is one billionth of a meter. These particles can range in size from 1 to 100 nanometres, making them significantly smaller than a human hair (Santoshkumar, Rajeshkumar & Kumar 2017). What is extraordinary about nanoparticles is that their characteristics and behaviour may differ substantially from the same substance in bulk form. This is because in the nanoscale, particles have a higher surface area than volume, resulting in unique physical, chemical, and biological properties (Bordiwala, 2023). Nanoparticles can take the form of spheres, rods, tubes, or even more complicated structures. Nanoparticles can be synthesized using different methods, such as chemical, sol-gel, thermal decomposition, and green methods. Among these, the green method stands out due to its environmentally friendly nature, cost-effectiveness, biocompatibility, and ability to produce nanoparticles with unique properties. As a result, green methods are increasingly gaining attention from researchers. Because of their tiny size, they have amazing qualities such as improved optical, electrical, magnetic, and catalytic capabilities. Because of these characteristics, nanoparticles are extremely adaptable and beneficial in a wide range of applications (Nguyen et al., 2023). Nanoparticles have gained significant attention in various fields, including environmental science, due to their potential applications and benefits. Nanoparticles have a wide range of applications in various fields. They are utilized in water treatment to eliminate contaminants, where iron nanoparticles capture heavy metals and activated carbon nanoparticles address organic pollutants. In the realm of air pollution control, photocatalytic nanoparticles like titanium dioxide break down harmful substances like nitrogen oxides and volatile organic compounds when exposed to light (Punia et al., 2021). Nanoparticles also play a crucial role in improving energy generation and storage systems. In solar cells, they enhance light absorption, electron transport, and conversion efficiency, while in batteries and fuel cells, they contribute to enhanced performance (Sotoudeh et al., 2023). Waste management benefits from nanoparticles

as well, as they assist in the decomposition of organic waste and remediation of contaminated sites. Certain nanoparticles possess enzymatic or catalytic properties that enable efficient waste treatment. Moreover, in agriculture, nanoparticles boost crop productivity, reduce reliance on chemical fertilizers and pesticides, and minimize environmental impact through targeted delivery of nutrients and agrochemicals (Rani et al., 2023).

Nickel Oxide (NiO)

Nickel oxide is a compound composed of nickel and oxygen atoms, with the chemical formula NiO. It is a dark green or black solid material that can be found naturally as the mineral bunsenite. The semiconducting behaviour of nickel oxide is one of its most prominent properties. It has both metallic and insulating qualities, making it valuable in electronics and optoelectronics (Venkatalakshmi, Kini & Naik 2023). Nickel oxide is often employed as a p-type semiconductor material, which means it may conduct positive charge carriers (holes) when properly doped. Nickel oxide (NiO) is a semiconductor made up of nickel and oxygen atoms. Because of its metallic and insulating qualities, it can be used in electronics and optoelectronics. Nickel oxide is used in resistors, capacitors, magnetic sensors, and solid oxide fuel cells as a cathode material. It's used as a catalyst in a variety of chemical processes, such as oxidation, hydrogenation, and carbon dioxide reforming. Nickel oxide is also being investigated for prospective use in energy storage systems such as lithium-ion batteries, super capacitors, and fuel cells. Its distinct qualities make it useful in electronics, catalysis, and energy storage (Ahmad et al., 2022).

Copper Oxide (CuO)

Any substance containing copper and oxygen is referred to as copper oxide. Copper oxide is classified into two types: copper (I) oxide (Cu_2O) and Copper (II) oxide (CuO), each having its own set of chemical characteristics and applications. Cu_2O (Copper (I) oxide) is a crimson or reddish-brown chemical. It occurs naturally as the mineral cuprite, although it may also be synthesized. Copper oxide is a p-type semiconductor (Waris et al., 2021). Copper (I) oxide is a popular pigment used in ceramics, glass, and paints. It is also employed in the manufacture of solar cells, as a catalyst in certain chemical processes, and in the fabrication of semiconductors. Copper (II) oxide (CuO): A dark substance. It is found in nature as the mineral tenorite and may be created by melting copper in the presence of oxygen. Copper (II) oxide is mostly utilized as a raw

material in the manufacture of various copper compounds. It is also used as a pigment, a catalyst, and in organic chemical synthesis (Moudgil et al., 2022).

Aegle Marmelos

Aegle marmelos, often known as bael or bael fruit, is a tree endemic to the Indian subcontinent and Southeast Asia. It is a member of the Rutaceae family and is well-known for its medicinal and nutritional benefits. Various elements of the *Aegle marmelos* tree, including the fruit, leaves, bark, and roots, have traditionally been utilized for medicinal purposes in Ayurvedic and traditional medicine systems. Bael plant is dense in vitamins, minerals, and antioxidants and is therefore very nutritious. It includes vitamins C, A, B complex, potassium, calcium, iron, and fibre, among other things. Carotenoids, phenolics, alkaloids, pectin, tannins, coumarins, flavonoids, and terpenoids are among the many bioactive components found in bael (Baliga et al., 2011).

Literature Review

Present study reported that Copper oxide nanoparticles were synthesized using a simple, inexpensive, and environmentally friendly method, utilizing an extract from *Cedrus deodara* as a safe reducing agent. X-ray diffraction analysis revealed that the nanoparticles possessed a monoclinic structure. Furthermore, examination under scanning electron microscopy and transmission electron microscopy confirmed that the nanoparticles exhibited a spherical shape with consistent symmetry. To assess their potential as antibacterial agents, the CuO nanoparticles were subjected to the disc diffusion method against *E. coli* and *S. aureus*. Remarkably, the synthesized nanoparticles displayed significant antibacterial activity, particularly against *E. coli*, with an impressive maximum inhibition zone of 29 mm (Ramzan et al., 2021).

In this study, copper oxide nanoparticles were synthesized using *Eucalyptus Globulus* leaf extract. The nanoparticles had a spherical shape with an average size of 88 nm and a negative zeta potential of -16.9 mV, as confirmed by SEM and DLS analysis. These nanoparticles showed a high adsorption capacity for methyl orange dye, with an efficiency of 95 mg/g at room temperature. Optimal adsorption occurred at pH 4.5 with a dye quantity of 0.04 g/50 mL. The Box-Behnken design in response surface methodology successfully optimized the process parameters, and the model used was deemed appropriate with an R^2 value of 0.99. Overall, the study demonstrated the effective decolorization of methyl orange using the synthesized copper oxide nanoparticles (Alhalili Zahrah, 2022).

In the present study, copper oxide nanoparticles (CuO NPs) were synthesized using an aqueous extract of *Calotropis procera*. The nanoparticles exhibited a highly crystalline structure, with sizes ranging from 40 to 100 nm, as determined by XRD analysis. SEM and TEM images confirmed the spherical shape of the nanoparticles, with sizes ranging from 20 to 80 nm. The anti-fungal and anti-bacterial activities of the synthesized CuO NPs were evaluated. At a concentration of 100 µg/mL, significant inhibition of both phytopathogens was observed in both in vitro and detached leaf assays of Muskmelon (Shah et al., 2022).

This study reported the synthesized copper oxide nanoparticles (CuO NPs) using petals from *Rosa delicia* (R.d), *Rosa kardina* (R.k), and *Rosa foetida* (R.f). R.k-CuO NPs exhibited the highest antioxidant capacity (98.700.3 µg AAE/mg) and reducing power potential (73.50.18 µg AAE/mg), along with the greatest DPPH scavenging potential (60.9%). R.d-CuO NPs and the extract demonstrated strong inhibition of urease, while R. f-CuO NPs and R.k-CuO NPs showed notable inhibition of α-amylase and lipase, respectively. Remarkably, both R.k-CuO NPs and R. f-CuO NPs displayed significant peroxidase-like activity (Asghar et al., 2022).

This study presented that the primary objective was to synthesize copper oxide nanoparticles (CuONPs) using *Canthium coromandelicum* leaves, employing a cost-effective, simple, and sustainable method. The photocatalytic assessment demonstrated that the synthesized CC-CuONPs exhibited a remarkable degradation efficiency of 91.32% for methylene blue (MB) and 89.35% for methyl orange (MO). These results suggest that the biosynthesized CC-CuONPs have the potential to effectively eliminate contaminants in an environmentally friendly manner (Selvam et al., 2022).

Present study reported the aim to synthesize copper oxide nanoparticles (CuONPs) using *Mangifera Indica* aqueous extract as a biologically derived reducing agent. The synthesized CuONPs were utilized as a photo-catalyst for the degradation of malachite green under visible light. The remarkable bandgap energy of 1.62 eV contributed to the CuONPs excellent performance, achieving a high degradation efficiency of 95.39%. The CuONPs also demonstrated strong durability over five cycles of recycling. Additionally, the CuONPs exhibited significant antibacterial activity against both *E. coli* and *S. aureus* strains under light conditions, outperforming their activity in darkness (Dat et al., 2023).

In a conducted investigation, copper oxide nanoparticles were synthesized utilizing leaf extract derived from *Spinacia oleracea* (spinach). The synthesized CuO nanoparticles

displayed notable bactericidal properties against *Staphylococcus aureus*, *Escherichia coli*, *Proteus*, and *Klebsiella*. Furthermore, they exhibited promising antifungal activity against *A. flavus, Rhizopus,* and *A. fumigant*. Additionally, the CuO nanoparticles demonstrated significant larvicidal activity against *Culex Quinquefasciatus* mosquitoes (Thandapani et al., 2023).

Present study reported the synthesis of CuO nanoparticles (CuO NPs) using a one-pot synthetic method that involves microwave irradiation and the utilization of apple peel extract as a safe reducing and capping agent. The synthesized CuO NPs exhibited excellent performance in reducing MB dyes and CV dyes. Moreover, the catalysts can be easily recovered and reused without significant loss of activity (Rajamohan R, Lee & Y.R 2023).

Present study reported that the NiCl2 and *Tinosphora cordifolia* stem extract were used in an ultrasound-assisted process to create NiO nanoparticles (NiO NPs). A hydrothermal technique was used to distribute these NPs into rGO powder. The results showed that the NiO/rGO composite performed exceptionally well as a photocatalyst. When exposed to both UV and visible light, it demonstrated outstanding photocatalytic activity in degrading tetracycline, attaining full elimination in 30 minutes. The presence of rGO contributed considerably to photocatalytic activity, as demonstrated by a larger turnover number (6.67) compared to NiO NPs (0.55 and 0.41 under UV and visible light, respectively). Furthermore, the antibacterial activities of NiO/rGO against *Staphylococcus aureus* and *Escherichia coli* were proven by its low minimum inhibitory concentrations (MIC) (Fatimah et al., 2023).

This study reported that NiO nanoparticles were synthesized using orange fruit waste. Analysis with SEM and DLS revealed that the nanoparticles exhibited cubic and triangular shapes, with sizes ranging from 21 nm to 130 nm. Notably, these orange fruit waste-derived NiO nanoparticles displayed excellent antimicrobial activity against common microbial pathogens, with the most pronounced effect observed against *E. coli*, followed by *A. niger*, *K. pneumoniae*, *B. subtilis*, and *S. aureus*, when tested at a concentration of 200 μg mL^{-1}. Additionally, the synthesized nanoparticles demonstrated significant scavenging activity against DPPH, OH, and FRAP free radicals (Narayanan et al., 2023).

A research study demonstrated the synthesis of NiO nanoparticles using leaf extract from *Ananas comosus* (pineapple). The photocatalytic properties of these nanoparticles were explored by investigating the degradation of low-density polyethylene (LDPE)

film in their presence. The results revealed that the polymeric nanocomposites (NCs) experienced a substantial weight loss of $33 \pm 1.6\%$ when exposed to solar light for 240 hours, while pure LDPE only showed a weight reduction of $8.6 \pm 0.7\%$ under the same conditions. In contrast, the nanocomposites exhibited a weight reduction of $1.90 \pm 0.05\%$ after 240 hours in the absence of light (Olajire AA & Mohammed AA 2023).

A study documented the synthesis of NiO nanoparticles using an aqueous extract of *Senna auriculata* flowers. The antibacterial properties of these nanoparticles, produced through a biological process, exhibited greater efficacy against Gram-negative bacteria such as *Escherichia coli* when compared to Gram-positive microorganisms. Additionally, the catalytic activity of the nanoparticles was examined for the degradation of methylene blue (MB) dye under direct visible light. The findings demonstrated an impressive degradation efficiency of 97% for the MB dye after a 90-minute exposure (Al-Zaqri et al., 2022).

This paper presents the synthesis of Nickel oxide nanoparticles (NiO NPs) using *Solanum trilobatum* leaf extract as the fuel and nickel nitrate as the precursor, utilizing the hot plate combustion method. The as-synthesized NiO nanomaterial was evaluated for its cytotoxic activity on A549 cell culture, antibacterial activity against both gram-positive and gram-negative bacterial pathogens, and its ability to photo catalytically degrade 4-Chlorophenol (4-CP), an endocrine-disrupting chemical. The synthesized NiO nanoparticles demonstrated excellent performance against A549 cells, effective antibacterial activity against gram-positive pathogens, and exhibited photocatalytic activity by efficiently degrading 4-Chlorophenol under UV light, as confirmed by TOC experimental results (Ezhilarasi et al., 2020).

This study focused on the eco-friendly synthesis of nickel/nickel oxide nanoparticles (Nio/NiO nanoparticles) using the extract derived from *Lactuca Serriola* seeds. The antibacterial activity of these nanoparticles was also assessed against eight pathogenic bacteria, including P*seudomonas aeruginosa, Staphylococcus aureus, Staphylococcus epidermidis, Basilus subtilis, Basilus pumilus, Micrococcus luteus, E. coli*, and *Bordetella bronchiseptica*. The results demonstrated significant antibacterial effects at low doses, with nearly complete inhibition observed at the optimized concentration of nanoparticles (Ali et al., 2022).

Nickel oxide nanoparticles were synthesized using a green microwave combustion method. The XRD analysis confirmed the high crystallinity of the material, with an

average crystallite size of 20 nm and 30 nm for the two NiO samples. The SEM analysis revealed that the samples had a spherical shape and exhibited some agglomeration. The antibacterial study demonstrated that both samples displayed effective antibacterial activity against both gram-positive and gram-negative bacteria. Thus, these environmentally friendly synthesized NiO nanoparticles have potential applications in antibacterial treatments (Ghazal et al., 2021).

Research Gap Identification

There is limited literature available on the synthesis of Nickel oxide (NiO) and Copper oxide (CuO) using *Aegle marmelos* plant extract at specific temperatures. This study aims to investigate and enhance the properties of NiO and CuO nanoparticles synthesized through a green approach. Furthermore, the synthesized nanostructures will be explored for their potential environmental applications.

Objectives

Based on the aforementioned discussion, the current study aims to achieve the following objectives:

- To investigate the synthesis of nickel oxide and copper oxide nanostructures using a co-precipitation method, by utilizing an extract derived from the *Aegle marmelos* plant for the green synthesis process.
- To characterize by using various techniques including X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), UV-Visible spectroscopy, scanning electron microscopy (SEM), transmission electron microscopy (TEM).
- To study the environmental applications of synthesized nanostructures.

Proposed Methodology

Materials

Nickel nitrate hexahydrate (Ni $(NO_3)_2.6H_2O$), Sodium hydroxide (NaOH) pellets, Copper (II) nitrate trihydrate (Cu $(NO_3)_2.3H_2O$), The leaves of the *Aegle marmelos* plant will be gathered from various locations within Hamirpur district, Himachal Pradesh, to synthesize a plant extract.

Synthesis of *Aegle marmelos* plant extract

To start the process, fresh leaves of the *Aegle marmelos* plant will be gathered from local areas in Hamirpur, Himachal Pradesh, India. These leaves will undergo thorough washing using both tap water and distilled water to eliminate any dust, microorganisms, or impurities present on them. An accurate amount of 10 grams of the washed leaves will then be weighed and placed in a mortar. The leaves will be finely chopped and

crushed in the mortar. Subsequently, the minced leaves will be boiled in 100 mL of distilled water at a temperature of 80 °C for approximately 30-35 minutes. This boiling process will be carried out using an assembly that involves attaching a condenser to a round bottom flask, which will be placed on a Rota-mantle for constant stirring. After boiling, the extract will be allowed to cool and then filtered through a Whatman filter paper. The resulting filtered extract solution will serve as the basis for synthesizing nanostructures of NiO and CuO.

Synthesis of NiO and CuO nanostructures

The co-precipitation method will be utilized to synthesize NiO and CuO nanostructures. In this process, nickel nitrate hexahydrate and copper nitrate trihydrate will serve as the precursor materials. To enable a green synthesis approach, a plant extract will be employed as a surfactant. The synthesis procedure begins by dissolving 0.05 M of nickel nitrate or copper nitrate in 50ml of distilled water. Subsequently, the plant extract is added drop by drop to the prepared solution. Next, a 1 M NaOH solution is introduced to the mixture. The resulting aqueous solution is then stirred for 2 hours at a temperature of 80 ^{o}C. The nanoparticles obtained from the solution are filtered and subjected to multiple washes with distilled water and ethanol. Afterward, they are dried in a vacuum oven at 60 ^{o}C for 12 hours. Finally, the dried nanoparticles undergo calcination at a temperature of 400 ^{o}C for duration of 3 hours.

Characterization of synthesized NiO and CuO nanostructures

Several techniques were employed to thoroughly analyze the characteristics of the synthesized nanostructures. X-ray diffraction (XRD) was utilized to measure the angle of diffraction within a specified range. The surface morphology was evaluated using a high-resolution scanning electron microscope (SEM) equipped with energy dispersive analysis. The size and shape of the sample were estimated using high-resolution transmission electron microscopy (TEM). Fourier transform infrared spectroscopy (FTIR) was used to examine the mineral composition and physical properties and UV-visible spectroscopy was used to measure the sample's absorbance.

Thesis Outline

The thesis will be comprised of following chapters:

Chapter 1 (Introduction): This chapter provides a concise overview of nanotechnology, highlighting the characteristics of NiO and CuO nanostructures, as well as the potential of Aegle marmelos plant extract. Additionally, it outlines the objectives of the proposed study.

Chapter 2 (Literature Review): This section will focus on a comprehensive exploration of profound literature concerning the synthesis, characterization, and usage of nanostructures made of NiO and CuO. Additionally, it will address the identification of gaps in existing research.

Chapter 3 (Materials and Methods): This section will provide a comprehensive discussion on the properties of NiO and CuO nanostructures, including their composition, environmentally friendly production techniques, characterization methodologies, and various applications.

Chapter 4 (Results ad Discussion): This section will present the analysis of the findings from the research conducted on the physical and chemical characteristics of nanostructures of NiO and CuO. The chapter will contain detailed reports on the properties observed, followed by an in-depth analysis and interpretation of the results.

Chapter 5 (Summary and Conclusion): In conclusion, this chapter provides a comprehensive summary of the entire study, highlighting its relevance to the stated objectives.

Chapter 6 (References): In this section, the cited sources will adhere strictly to the Harvard referencing style.

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CERTIFICATE
(SYNOPSIS APPROVAL CERTIFICATE)

This is to certify that the Synopsis for Ph.D. Thesis titled, **"ELUCIDATING THE SYNTHESIS, CHARACTERIZATION AN D VERSATILE APPLICATIONS OF NANOSTRUCTURES COMPRISING NICKEL OXIDE AND COPPER OXIDE"**, submitted by **Ms. Kashama Sharma** to the Career Point University, Hamirpur (H.P.), India in partial fulfilment of the requirements for the degree of **DOCTOR OF PHILOSOPHY** in the Department of **Chemistry**, School of **Basic and Applied Sciences** has been approved by the Research Advisory Committee (RAC) after an Oral Examination of the student on dated 17.06.2023.

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